भारतीय मानक
Indian Standard

IS 7704 : 2022

4 – अमिनोटोल्यून-3- सल्फोनिक एसिड — विशिष्टि

(*दूसरा पुनरीक्षण*)

4-Aminotoluene-3-Sulphonic Acid — Specification

(*Second Revision*)

ICS 71.080.40

भारतीय मानक ब्यूरो
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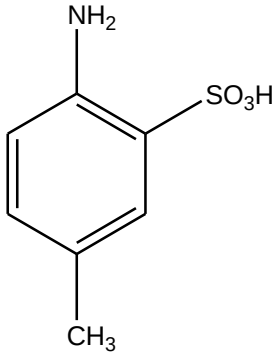
August 2022 **Price Group 6**

Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

4-Aminotoluene-3-sulphonic acid ($C_7H_9NO_3S$) is an important dye-intermediate widely used in the manufacture of dyes and pigments. It is also known as *p*-toluidine-*m*-sulphonic acid or 4B acid. It has the following structural formula:



4-AMINOTOLUENE-3-SULPHONICACID

IPUAC Name:2-amino-5-methylbenzenesulphonic acid

[MOLECULAR MASS: 187]

(CAS REGISTRY NO.:88-44-8)

This standard was first published in 1975 and subsequently revised in 1993. In the first revision the requirement of assay and 4-aminotoluene-2,5-disulphonic acid was modified in the light of experience gained. Besides, a maximum requirement for matter insoluble in sodium carbonate solution was included.

In this (*second*) revision, HPLC method for determination of assay of 4-aminotoluene-3-sulphonic acid has been incorporated.

The composition of the Committee responsible for formulation of this standard is given in Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

4-AMINOTOLUENE-3-SULPHONIC ACID — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and testing for 4-aminotoluene-3-sulphonic acid.

2 REFERENCES

The following standard contain provisions, which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification *(third revision)*
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of light brown powder. It shall be free from dust and other visible impurities.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 of Table 1.

Table 1 Requirements for 4-Aminotoluene-3-sulphonic acid

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.2)

Sl No.	Characteristics	Requirement (on dry basis)	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Assay by nitrite value, percent by mass, *Min*	98	A
	Or		
	Assay by HPLC[1)], percent by mass, *Min*	98	B
ii)	4-Aminotoluene, percent by mass, *Max*	0.5	C
iii)	4-Aminotoluene-2,5-disulphonic acid, percent by mass, *Max*	0.5	D
iv)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	0.3	E

NOTES

1 In case of disputes, determination of assay by HPLC, shall be the referee method.

2 The thin layer chromatography (TLC) method (in Annex D) for determining of impurity, 4-aminotoluene-2,5-disulphonic acid is to be performed only when this impurity is available in its pure form.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

4.2.1 Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of the material;
d) Batch or lot number;
e) Month and year of the manufacturer; and
f) Any other statutory requirement.

4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Test for determination of assay shall be conducted on individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, 4-aminotoluene, 4-aminotoluene-2,5-disulphonic acid and matter insoluble in sodium carbonate solution shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of assay if each of the individual test results as obtained in **5.2.1** satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Sample*

The lot shall be declared as conforming to the requirements of 4-aminotoluene, 4-aminotoluene-2,5-disulphonic acid and matter insoluble in sodium carbonate solution, if the test results satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 PREPERATION OF SAMPLE

Dry the material at 105 ± 1 °C to constant mass. Grind and mix well. Transfer the material to a wide mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for test.

6.2 Tests shall be carried out on the prepared sample (**6.1**) according to the methods as prescribed in col 4 of Table 1.

6.3 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF 4-AMINOTOLUENE-3-SULPHONIC ACID (ASSAY) BY NITRITE VALUE

A-l OUTLINE OF THE METHOD

An accurately weighed quantity of the material is dissolved in hydrochloric acid and water and the total amine is determined by diazotization with a standard sodium nitrite solution at 0–5 °C.

A-2 APPARATUS

A-2.1 Magnetic Stirrer with Rotor

A-3 REAGENTS

A-3.1 Standard Sodium Nitrite Solution, 0.2 N (freshly standardized).

A-3.2 Starch Potassium Iodide Paper

A-3.3 Hydrochloric Acid, approximately 30 percent (*m/v).*

A-3.4 Solid Potassium Bromide, reagent grade.

A-4 PROCEDURE

Weigh accurately 10.000 g of the prepared sample (**6.1**) and transfer to a beaker containing 400 ml water and 50 ml of hydrochloric acid. Stir to dissolve completely. Transfer to a 1 000 ml measuring flask and adjust to the mark with water. Transfer 100 ml of the solution accurately by means of a pipette into a 1 000 ml beaker containing 200 g of crushed ice and 200 ml of water. To this add 5 g of potassium bromide and 20 ml hydrochloric acid. Titrate with standard sodium nitrite solution with constant stirring maintaining the temperature between 0 °C to 5 °C throughout the titration. Carry out the titration as rapidly as possible until a distinct blue ring on the starch potassium iodide paper indicates the end point. The end point should persist for 5 min without further addition of the nitrite solution.

A-5 CALCULATION

$$\text{Assay, percent by mass} = \frac{V \times N \times 187}{M}$$

where

V = volume or standard sodium nitrite solution required for complete diazotization, ml;

N = normality of standard sodium nitrite solution; and

M = mass of the prepared sample taken for the test, g.

ANNEX B

[*Table* 1, *Sl No* (ii)]

DETERMINATION OF 4-AMINOTOLUENE-3-SULPHONIC ACID CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

B-1 GENERAL

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-2 APPARATUS

B-2.1 Binary Gradient Liquid Chromatography System, capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2.2 Column

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

B-3 REAGENT

B-3.1 Acetonitrile, HPLC grade.

B-3.2 Water, HPLC grade.

B-3.3 Disodium Hydrogen Orthophosphate

B-3.4 Ammonium Dihydrogen Orthophosphate

B-3.5 4-Aminotoluene-3-sulphonic Acid

B-4 STANDARD PREPARATION

Weigh accurately 0.050 0 g 4-aminotoluene-3-sulphonic acid in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

B-5 SAMPLE PREPARATION

Weigh accurately 0.050 0 g sample in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

B-6 BUFFER PREPARATION

Take 0.575 0 g ammonium dihydrogen orthophosphate and 0.700 0 g disodium hydrogen orthophosphate in 1 l volumetric flask. Add 200 ml HPLC grade water and completely dissolve it. Make the total volume with HPLC grade water.

B-7 FLOW RATE, 1.00 ml/min.

B-8 MOBILE PHASE

Acetonitrile	Buffer
25	75

B-9 COLUMN OVEN TEMPERATURE, 40 °C.

B-10 INJECTION VOLUME, 10 µl.

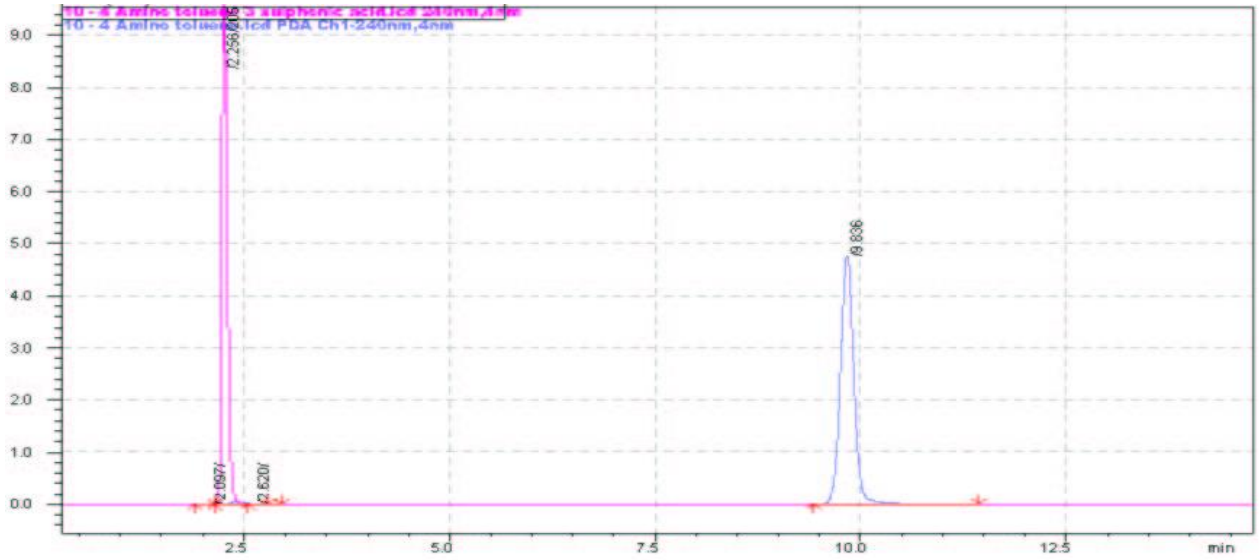
B-11 RUN TIME, 15 min.

B-12WAVELENGTH, 254 nm.

B-13 PEAK TIME

4-Aminotoluene-3-sulphonic acid: 2.25 min

4-Aminotoluene: 9.83 min



B-14 CALCULATION

Calculate the peak area of individual constituent pertaining to 4-aminotoluene-3-sulphonic acid on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure 4-aminotoluene-3-sulphonic acid.

$$4-\text{Aminotoluene}-3-\text{sulphonic acid, percent} = \frac{A_2 \text{ x } V_1 \text{ x } W_1 \text{ x } B_2}{A_1 \text{ x } V_2 \text{ x } W_2 \text{ x } B_1} \text{ x } 100$$

where

A_1 = area of standard 4-aminotoluene-3-sulphonic acid;

V_1 = injection volume of standard 4-aminotoluene-3-sulphonic acid;

W_1 = weight of standard 4-aminotoluene-3-sulphonic acid;

B_1 = total volume of standard 4-aminotoluene-3-sulphonic acid;

A_2 = area of 4-aminotoluene-3-sulphonic acid peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample

ANNEX C

[*Table* 1, *Item* (iii)]

DETERMINATION OF 4-AMINOTOLUENE IN 4-AMINOTOLUENE-3-SULPHONIC ACID

C-l GENERAL

Free 4-aminotoluene in 4-amiotoluene-3-sulphonic acid is determined by thin layer chromatography (TLC).

C-2 APPARATUS

C-2.1 Thin Layer Chromatographic Plates

Glass plate of size 20 cm × 20 cm coated uniformly with silica gel H or an equivalent powder and freshly activated for about 1 h at 90 °C.

C-2.2 Micropipette, 10 µl capacity.

C-2.3 Developing Chamber

C-2.4 Chromatographic Sprayer

C-3 REAGENTS

C-3.1 4-Aminotoluene, pure.

C-3.2 4-Aminotoluene-3-sulphonic Acid, pure.

C-3.3 Sodium Hydroxide Solution, 1 N.

C-3.4 Hydrochloric Acid Solution,1 N.

C-3.5 Dimethyl Formamide

C-3.6 Developing Solvent, Benzene: Methanol [8:2, (*v/v*)].

C-3.7 Spray Solution

A 0.2 percent solution of 1-naphthyl-ethylenediamine dihydrochloride in 1:1 dimethylformamide and 4 N hydrochloric acid.

C-4 PROCEDURE

C-4.1 Weigh accurately 1.0 g of the sample and dissolve it in 20 ml of 1 N sodium hydroxide solution by gentle warming. Cool and transfer to a 100 ml volumetric flask and make it up to the mark with methanol.

C-4.2 Weigh accurately 1.0 g of the pure 4-aminotoluene-3-sulphonic acid and dissolve it in 20 ml of 1 N sodium hydroxide solution. Transfer this to a 100 ml volumetric flask and add 5.0 ml of 0.1 percent solution of 4-aminotoluene in methanol and make it up to the mark with methanol.

C-4.3 Spot 10 µl each of the sample solution (**C-4.1**) and the reference solution (**C-4.2**) on the thin layer chromatographic plate. Dry well with the help of an air drier. Place the developer in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in ascending manners to run a distance of about 12 cm past the starting line. Remove the plate from the chamber and dry off the solvent in an air drier. Hold the plate for 20 s in nitrous acid fumes (formed by adding about 5 g of sodium nitrite to 50 ml of 5 N hydrochloric acid in a 5 l beaker). Allow the plate to stand for 1 min to remove excess nitrous fumes. Spray with the spray solution. After a few minutes examine visually the intensity of colour developed with the sample solution and compare it with that developed with a reference solution.

ANNEX D

[*Table* 1, *Item* (iv)]

DETERMINATION OF 4-AMINOTOLUENE-2,5-DISULPHONIC ACID CONTENT

D-l GENERAL

The estimation of 4-aminotoluene-2,5-disulphonic acid is done by thin layer chromatography (TLC) and this estimation of 4-aminotoluene-2,5-disulphonic acid, however, should be done only when pure form of this impurity is available.

D-2 APPARATUS

D-2.1 Thin Layer Chromatographic Plate

Glass plates of size 20 cm x 20 cm coated uniformly with silica gel H or an equivalent powder and freshly activated for about 1 h at 90°C

D-2.2 Micropipette, 10 µl capacity.

D-2.3 Developing Chamber

D-2.4 Chromatographic Sprayer

D-3 REAGENTS

D-3.1 4-Aminotoluene-3-sulpnonic Acid, pure.

D-3.2 4-Aminotoluene-2,5-disulphonic Acid, pure.

D-3.3 Developing Solvent

Methanol/benzene/methylisobutyl ketone/water (5:5:5:1).

D-3.4 Spray Solution

A strong ammonical solution of H-acid [0.5 percent (*m/v*)] of H-acid dissolved in 10 percent (*v/v*) ammonia solution.

D-4 PROCEDURE

D-4.1 Weigh accurately 1.0 g of the prepared sample (**6.1**) and dissolve it in 50 ml of a mixture of water and methanol (1:1) by warming on a water-bath. Cool, transfer it to a 100 ml flask with a water-methanol mixture (1:1).

D-4.2 Weigh accurately 1.0 g of pure 4-aminotoluene-3-sulphonic acid, dissolve as in **D-4.1**. Cool and transfer to 100 ml volumetric flask. Add 5.0 ml of 0.1 percent solution of 4-aminotoluene-2,5-disulphonic acid to the flask and dilute it to 100 ml with water-methanol mixture.

D-4.3 Spot 10 µl each of the sample solution (**D-4.1**) and the reference solution (**D-4.2**) on the thin layer plate. Dry well with the help of an air drier. Place the developer in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the mobile phase to run in the ascending manner to a distance of about 12 cm past the starting line. Remove the plate from the chamber and dry off the solvent in an air drier. Hold the plate for 20 s in nitrous acid fumes (formed by adding about 5 g of sodium nitrite to 50 ml of 5 N hydrochloric acid in a 5 l beaker). Allow the plate to stand for 1 min to remove excess nitrous fumes. Spray with a strongly ammonical solution of H-acid. After a few min examine visually the intensity of colour of each zone developed with the sample solution and compare it with that developed with a reference solution.

ANNEX E

[*Table* 1, Item (iv)]

DETERMINATION OF MATTER INSOLUBLE IN SODIUM CARBONATE SOLUTION

E-l REAGENTS

E-1.1 Sodium Carbonate Solution, 2 percent (*m/v*).

E-2 PROCEDURE

Weigh accurately 5 g of 4-aminotoluene-3-sulphonic acid and transfer it to a 500 ml beaker. Add 100 ml of 2 percent sodium carbonate solution. Heat the beaker to about 75-80 °C in a carbonate steam bath till most of the material is dissolved. Observe the opalescence. If the insoluble matter is more than a trace, filter through a previously washed, dried, and weighed sintered crucible of porosity G-4. Wash thoroughly the matter on the sintered crucible with water till alkali free, dry and weigh. Repeat the operation of drying and weighing until constant weight is obtained.

E-3 CALCULATLON

Calculate the percentage of alkali insoluble matter according to the formula:

$$\text{Matter insoluble in sodium carbonate solution, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass of residue; and

M = mass of material taken for the test

ANNEX F

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
Institute of Chemical Technology, Mumbai	PROF GANAPATI SUBRAY SHANKARLING (***Chairperson***)
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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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